Japanese Patent Laid-open No. Hei 2-255694

Laid-open: October 16, 1990

Title: A method of manufacturing molasses-containing crystals containing lactitol anhydride crystals and a method of manufacturing a molasses-containing crystal composition

Int. Class Number: (Int. Cl. 5) C 07 H 15/04

Application Number: Hei 1-75204

Application Date: March 29, 1989

Inventors: Shigeru Kawashima, Hiroshi Ide, Kazuaki Kato, Mitsuo Magara and Yoshifumi Ishii

Applicant: Towa Kasei Kogyo K.K.

Agent: Keiichi Ota, patent attorney

Claims:

1. A method of manufacturing molasses-containing crystals which contain lactitol anhydride crystals, characterized in that crystals of lactitol anhydride having a melting point of 150-155°C are separated from an aqueous solution of lactitol and are collected or solidified.

2. A method according to claim 1, wherein purity of the lactitol in the aqueous solution of lactitol is not lower than 95% by weight per died solid.

3. A method according to claim 1 or 2, wherein the concentration of the aqueous solution of lactitol is 90% by weight or higher.

4. A method according to claim 1, 2 or 3, wherein the temperature for separating lactitol anhydride crystals is 50-110°C.

5. A method according to claim 1, 2, 3 or 4, wherein seed crystals are made coexisted in the aqueous solution of lactitol at 40-100°C.

6. A method of manufacturing a molasses-containing crystal composition which contain lactitol anhydride crystals, characterized in that one or more selected from a group consisting of aspartame, saccharine, glycyrrhizin, acesulfame-K, stevioside, alpha-glycosylstevioside, dihydrochalcone, glycine and alanine is/are added to a mass kit containing lactitol anhydride or an aqueous solution of lactitol, followed by solidifying.

Detailed description of the invention:

(Technical Field)

The present invention relates to a method of manufacturing molasses-containing crystals which contain lactitol anhydride crystals and also to a method of manufacturing a molasses-containing crystal composition.

(Prior Art)

Lactitol is a compound wherein glucose moiety of lactose is reduced to sorbitol and is identical with 4-beta-D-glacto-pyranosyl-D-sorbitol.

Lactitol has been prepared, for example, in the following manner as described in Journal of Agricultural and Food Chemistry, Vol. 27, No. 4, pages 680-686 (1979) that an aqueous solution of lactose, concentration being 30-40% by weight, is hydrogenated at 100°C under 40 atm. of hydrogen, followed by removing the catalyst therefrom and purifying with iron exchange

resin or with activated carbon.

When the degree of sweetness of a 5% aqueous solution of sucrose is defined as 100%, the relative sweetness of lactitol of the same concentration is about 36% and is lower than those of sorbitol (relative sweetness: 65%) and of xzylitol (relative sweetness: 96%).

Incidentally, lactitol is hardly digested and absorbed in digestive organs and also hardly fermented by microorganisms in mouth. Accordingly, lactitol is quite suitable as the sweetening agent for low calorie food, diet food, food causing little caries and healthy food to those who are worrying about diabetes, obesity, adults diseases and carious tooth.

Further, aldehyde group is reduced in lactitol and, therefore, lactitol has high stability to heat and to chemicals such as alkali and can be advantageously used as a material for food and industrial materials.

Known methods for the manufacture of lactitol anhydride crystals are, for example, as described in Journal of American Chemical Society, Vol. 74, pages 1105 (1952) that they are crystallized from an alcoholic solution and, as described in Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences, Vol. 170, pages 47-50 (1920) that crystals of lactitol hydrate are dehydrated to obtain said crystals.

For use in food, there has been a demand for molasses-containing crystals wherein lactitol anhydride crystals of lower cost are contained and the molasses-containing crystal compositions. Conventional methods for manufacturing thereof

are that, after preparing a mass kit, it is spray-dried or that, after solidifying the mass kit by placing in a vat, the solid is cut, pulverized or dried.

(Problems to be solved by the invention)

However, the conventional methods of manufacturing lactitol anhydride crystals have several problems, for example, that the yield of crystals is low in a method wherein the crystals are separated from an alcoholic solution or molasses; that, when crystallization is repeated to increase the yield, there remains a crystal mother liquor or waste molasses of which purity is so low that separation of crystals from the alcoholic solution or molasses is no longer possible, causing disposal troubles; and that, in many cases, the alcohol comes to be in a mixed state with water and, therefore, recovery of the alcohol is difficult. In addition, as a result thereof, the manufacturing cost of the crystals is relative high.

When lactitol anhydride crystals are manufactured by drying mono-, di- or trihydrate of lactitol, it is necessary to conduct steps of drying, pulverizing, classifying, etc. after the hydrate crystals are manufactured. Among those, the drying step requires to adopt severe dehydrating conditions, e.g. relatively high temperature or relatively high degree of vacuum, whereby the step is long and troublesome. Thus, in this step, it is difficult to control moisture content, i.e. to adjust the degree of dehydration, and moreover, cost for the step is high.

On the other hand, there are also several disadvantages in the conventional methods of manufacturing molasses-containing

crystals which contain lactitol anhydride and of manufacturing molasses-containing crystal compositions.

One of such manufacturing methods is that a concentrated aqueous solution of lactitol is poured into a vat or the like, solidified by cooling and then cut, pulverized, dried and classified. However, in said method, the step of allowing the vat to cool for the solidification of said solution requires long period of time. Accordingly, wide space is required for the solidification and aging of half-finished products and, further, the half-finished products should be maintained in hygienic state because they are left to stand in the vat for a long period of time.

In a method by spray drying, there are many problems, too. In such a method, much cost is needed for the equipments and devices and, during operation, high energy is required, resulting in making products expensive. In addition, it is difficult to manufacture products with larger particle size.

Moreover, those methods do not always give the molasses-containing crystals wherein only lactitol anhydride crystals are contained, but, besides the desired lactitol anhydride crystals, lactitol monohydrate crystals, lactitol dihydrate crystals and a mixture thereof with compounds of other crystalline state or noncrystalline solid are often contained. Therefore, it is difficult to afford the product of uniform quality, whereby it is difficult to make the physicochemical properties of the product constant.

The attempt of adding a sweetening agent such as aspartame

for preparing the product in a form of a composition also has problems, because aspartame is not stable to heat, whereupon, in the manufacturing method by solidifying in a vat, aspartame is apt to be decomposed. In view of the above, the methods adopted are limited to an expensive spray drying method or the like.

However, in the spray drying method, the once manufactured lactitol powder is made in a fluid state in a spray drying device and, then, an aqueous solution or slurry of a sweetening agent such as aspartame is spraied thereon and dried. Consequently, steps of spray-drying, pulverizing and classifying should be duplicated, making the cost higher.

Under such circumstances, there has been a demand for developing a method of manufacturing molasses-containing crystals which contain lactitol anhydride crystals or compositions thereof, which are capable of being used in food or as industrial materials and are inexpensive, by simple steps suitable for mass production.

(Means to solve the problems)

In order to solve the above-given problems, the present inventors have conducted extensive studies for the manufacture of molasses-containing crystals which contain lactitol anhydride crystals or the manufacture of a composition of said molasses-containing crystals, and, by adopting the kneading method and selecting specific manufacturing conditions, they succeeded in manufacturing such crystals and the composition of such crystals directly from an aqueous solution of lactitol or said solution

containing, if necessary, other components, by efficient and simple steps without separation of molasses therefrom, and also succeeded in manufacturing the products continuously in efficient manner. On that basis, the present invention has been achieved.

Thus, the present invention relates to a method of manufacturing molasses-containing crystals which contain lactitol anhydride crystals and also to a method of manufacturing a composition of said molasses-containing crystals, characterized in that, lactitol anhydride crystals are separated from an aqueous solution of lactitol or one containing other composition components, followed by isolating or solidifying.

There is no particular limitation as to the manufacturing method and origin of the aqueous solution of lactitol to be used in the present invention, so far as its lactitol purity is 95% by weight or more and its concentration is 90% by weight or higher.

The method of the present invention may, for example, be actually carried out in the following manner.

First, an aqueous solution of not less than 90% by weight, preferably, 91-99% by weight, of lactitol with a purity of not lower than 95% by weight is heated to 80°C or higher, preferably, to 85-110°C.

When the concentration is less than 90% by weight, there is a possibility that, after this step, the molasses-containing crystals wherein lactitol anhydride crystals are contained may be contaminated with hydrate crystals, whereupon the properties

of the product will not be uniform.

When the heating temperature is lower than 80°C, there is a possibility that, in the aqueous solution, various crystal embryo of lactitol may remain, whereupon the final product may be contaminated with various forms of crystals of lactitol.

When purity of the lactitol to be used is lower than 95% by weight, it so happens in many cases that the molasses-containing crystals wherein lactitol anhydride crystals are contained or the molasses-containing crystal compositions become highly hygroscopic.

The aqueous solution of lactitol is then cooled, while mixing, in a device by which cooling and mixing are possible, to not more than 110°C, preferably to 60-100°C, immediately before seed crystals are added. When the temperature of the aqueous solution is at 40-100°C, preferably at 50-90°C, lactitol anhydride crystals are added as seed crystals, and the whole is kept at 50-100°C to separate and grow lactitol anhydride crystals.

It is not always necessary that the seed crystals used here are lactitol anhydride crystals, but molasses-containing crystals wherein lactitol anhydride crystals are contained may be used as well. Moreover, a composition containing said molasses-containing crystals will do, too. However, the use of seed crystals containing crystals other than lactitol anhydride crystals causes inconstant quality of molasses-containing crystals or the composition containing said molasses-containing crystals as the final product.

When the temperature at the adding step of the seed crystals

is lower than 50°C, degree of supersaturation of lactitol becomes too large and crystals may have been separated out previously, whereupon crystals other than lactitol anhydride crystals may be formed. When the temperature is higher than 100°C, the added seeds of lactitol anhydride crystals may be dissolved, whereupon the effect as the seeds will not be well achieved.

When the temperature for separation and growth of the crystals is lower than 50°C, there are some problems on operation, such as solidification of the aqueous solution, and moreover, a very powerful cooling device is necessary to control the temperature of the lactitol therein to lower than 50°C. Accordingly, that is not preferred from economical viewpoint. On the contrary, when the temperature is higher than 110°C, the product will get scorched smell or be colored depending upon the purity of the lactitol in the aqueous solution. In the case of the composition, the components other than lactitol may be decomposed.

It is preferred that the temperature of the slurry (mass kit) or powdery lactitol anhydride-containing molasses crystals extruded from the device after mixing is not higher than 80°C, because such a slurry or powder is suitably hard for forming and other processes and the temperature-rising thereafter can be prevented.

The time from addition of the seed crystals to extrusion, when the above device is used, is preferably 30 seconds to 10 minutes, more preferably, 1-5 minutes, in working the present invention.

When the time is shorter than 30 seconds, it may happen that the crystals do not grow well, while, when it is longer than 10 minutes, there will be no bad influence on the quality of the molasses-containing crystals but much cost is needed for power maintenance and, accordingly, is senseless from economical viewpoint.

The amount of the seed crystals of lactitol anhydride to be added is preferably 0.05-2.0 to one part by weight of the solids content in the aqueous solution of lactitol, in terms of effecting crystallization quickly.

There will be no particular restriction for the device in working the present invention, so far as it can well mix the slurry while cooling and crystallizing. Particularly preferred ones are, for example, commercially-available laboplast mill, ribbon mixer, monoaxial extruder and biaxial extruder, since they exhibit sufficient mixing power and because the cooling temperature and cooling rate are easily controllable in them.

By the method as described above, it becomes possible to manufacture molasses-containing crystals wherein lactitol anhydride crystals are contained or a composition of the molasses-containing crystals without production of other lactitol crystals than lactitol anhydride crystals, by far easier operation than that of the conventional molasses-separation methods, quickly and, if necessary, continuously.

With respect to one or more sweetening agent(s) selected from a group consisting of aspartamem saccharine, glycyrrhizin, acesulfame-K, stevioside, alpha-glycosylstevioside, dihydro-

chalcone, glycine and alanime to be employed in the composition in the present invention, there will be no particular specification for their manufacturing method and origin, so far as they meet the official standards for foods and food additives when they are used in food, and, when they are used as medical or industrial materials, they meet the official standards for medical supplies and industrial materials.

With respect to the conditions for working the present invention, it is preferred to adopt the conditions of as mild as possible for prevention of decomposition of those substances. Particularly, when the sweetening agent to be mixed is aspartame, it is preferred to carry out the working at 50-95°C for 30 seconds to 10 minutes.

Working of the present invention under such conditions brings advantages that the product does not contain crystals of mono- and dihydrates of lactitol, that the steps can be completed within short period of time, and that no filtrate is formed, which further bring the advantages such as narrow temperature range for melting, improved yield and a low degree of decomposition of the composition.

(Examples)

The present invention will be further illustrated by way of the following examples wherein % is based on weight unless otherwise stipulated.

Example 1

65 grams of an aqueous solution of lactitol (purity=99.1%; concentration=98%) was placed in a Plaboplast mill at 95°C, 17

g of crystals of lactitol anhydride were added thereto at 80°C, and the mixture was kneaded at 40 rpm for 3 minutes to obtain about 80 g of molasses-containing crystals wherein lactitol anhydride was contained. The crystals were dried in a shelf-type drier of 80°C for 100 minutes and the melting point of the dried crystals was measured to be 154°C.

The product has relatively low hygroscopicity and good sweet taste. Therefore, it can be advantageously used as a sweetening agent, a quality-improving agent or a chemical-industry material for various types of beverages, foods, cosmetics and medical supplies.

Example 2 (Manufacturing example)

An aqueous solution of lactitol (purity=98.0%; concentration=96.4%) was continuously supplied at the rate of 15 kg/hr to a biaxial extruder for crystallization wherein the internal temperature of the first stirring part was previously adjusted to 85°C, and forwarded to the second stirring part with stirring at the rate of 80 rpm. Lactitol anhydride crystals as seed crystals were continuously added thereto at the rate of 2.5 kg/hr from an opening of the second stirring part which was cooled to 80°C, and the whole was forwarded to the third and the fourth stirring parts with stirring. The temperature of the substance in the third and the fourth stirring parts was further cooled down to 70°C. The extruder was operated under the condition such that the product was extruded about two minutes after the addition of the seed crystals to give molassescontaining crystals which contain lactitol anhydride crystals.

The resulting molasses-containing crystals were dried under the same conditions as in Example 1, and the melting point was measured to be 153°C.

Example 3

The inner temperature of the biaxial extruder for crystallization was adjusted to 100°C, and an aqueous solution of lactitol (purity=99.0%; concentration=94%) was poured thereinto at the rate of 16 kg/hr, then send to the second stirring part with stirring at 60 rpm. The second stirring part was cooled to 80°C. The molasses-containing crystals which contain lactitol anhydride crystals obtained in Example 2 were added thereto at the rate of 2 kg/hr as seed crystals, and the whole was sent to the third and the fourth stirring parts with stirring. The temperature of the substance in the third and the fourth stirring parts was further cooled to 75 to 60°C. After about 2 minutes from the addition of the seed crystals, the substance was extruded and formed into noodle-shape of 3-5 mm in diameter by a peletter equipped at the extruding part of the extruder and dried, pulverized and classified to give molasses-containing crystals which contain lactitol anhydride crystals.

Melting point of the resulting molasses-containing crystals was measured to be 155°C.

Example 4

The inner temperature of the biaxial extruder for crystallization was adjusted to 90°C, and an aqueous solution of lactitol (purity=99.0%; concentration=98%) was poured thereinto at the rate of 17.5 kg/hr, then sent to the second stirring part

with stirring at 60 rpm. The second stirring part was cooled to 70°C. Lactitol anhydride crystals were added thereto at the rate of 2.5 kg/hr as seed crystals, and, at the same time, a slurry of aspartame prepared by dissolving 1 part by weight of aspartame in 2 parts by weight of water was added at the rate of 20 g/min, and then the mixture was sent to the third and the fourth stirring parts. The third and the fourth stirring parts were cooled to 70 to 60°C. After about 2 minutes from the addition of the seed crystals, the mixture was extruded and formed into noodle-shape of 3-5 mm in diameter and dried, pulverized and classified to give a molasses-containing crystal composition wherein lactitol anhydride crystals were contained.

The resulting molasses-containing crystal composition exhibits about four times as much sweetness as sugar does and is a substantially low calorie sweetening agent with little hydroscopicity, good storability and substantially no caries-forming.

(Effect of the invention)

As fully illustrated hereinavobe, the present invention makes it possible to manufacture molasses-containing crystals wherein lactitol anhydride crystals are contained and compositions of said molasses-containing crystals, by a method suitable for mass production and, if necessary, in continuous manner, in which method the operation is simple, the operation time is short, hygienic maintenance is easy, cost for devices is relatively low and degree of decomposition of sweetening agents such as aspartame is low.